PCT





INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 3: (11) International Publication Number: A1 C12P 7/06

(43) International Publication Date:

11 May 1983 (11.05.83)

WO 83/01627

PCT/SE82/00369 (21) International Application Number:

(22) International Filing Date: 5 November 1982 (05.11.82)

(31) Priority Application Number:

8106611-0

(32) Priority Date:

6 November 1981 (06.11.81)

(33) Priority Country:

(71) Applicant (for all designated States except US): ALFA-LAVAL AB [SE/SE]; Box 500, S-147 00 Tumba (SE).

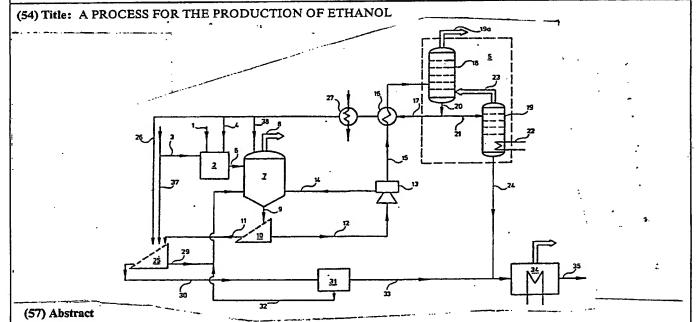
(75) Inventor/Applicant (for US only): WALLNÉR, Mats [SE/SE]; Östgötagatan 81 4 tr., S-116 64 Stockholm

(74) Agent: CLIVEMO, Ingemar; Alfa-Laval AB, Box 500, S-147 00 Tumba (SE).

(81) Designated States: DE, FR (European patent), US.

Published

With international search report. With amended claims.



In a process for the production of ethanol through continuous fermentation of a carbohydrate containing substrate which contains fermentable substance as well as non-fermentable soluble and solid substance, the substrate is hydrolyzed in a hydrolysis step (2) and fermented in a fermentor (7) comprised in a continuous process circuit comprising the fermentor (7), a yeast separation step (13) and a distillation step (5). From the fermentor a stream (9) of fermentation liquor is continuously withdrawn, and is then separated into a stream (11) enriched in solid substance, which is discharged from said process circuit, and a stream (12) impoverished in solid substance, which is fed to said yeast separation step (13). From the yeast separation step a yeast concentrate stream (14) is recirculated to the fermentor and the yeast-free stream i sent to the distillation step (5), wherein ethanol is distilled off and a remaining stillage stream (17) is obtained, part of which is continuously recirculated to the fermentor or to the hydrolysis step (2).

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	LI	Liechtenstein
. AU	Australia	· LK	Sri Lanka
BE	Belgium	LU	Luxembourg
BR	Brazil	MC	Моласо
CF	Central African Republic	MG	Madagascar
CG	Congo	MR	Mauritania
CH	Switzerland	MW	Malawi
CM	Cameroon	NL	Netherlands
DE	Germany, Federal Republic of	NO	Norway
DK	Denmark .	RO	Romania
FI	Finland	SE	Sweden
FR	France	SN	Senegal
GA	Gabon	SU	Soviet Union
GB	United Kingdom	TD	Chad
HU	Hungary	TG	Togo
JP	Japan	US .	
KP	Democratic People's Republic of Korea		

A process for the production of ethanol

The present invention relates to a process for the production of ethanol by continuous fermentation of a carbohydrate containing substrate of a raw material, which except fermentable carbohydrates also contains cellulose fibres and/or other non-fermentable solid material. More specifically the invention relates to a further development of applicant's preceding patented pro-10 . cesses for continuous ethanol fermentation at preferably atmosphere pressure in a fermentor with continuous yeast recirculation and continuous stillage recirculation.

The Swedish patent application 7801133-5 discloses a 15 method for continuous ethanol fermentation at preferably atmospheric pressure with continuous yeast recirculation and continuous stillage recirculation. The most important advantages with this method are the possibilities to ferment a substrate that is very con-20 centrated in fermentable substance, the addition of minimum amount of water to the process, thereby minimizing the power requirement for distillative separation of the ethanol, and the production of a stillage of such a high concentration that the stillage, pre-25 viously constituting a waste disposal problem, now offering an essential positive contribution to the total process economy, for example as a raw material for animal feed stuff of high quality. Since the discharge of a concentrated stillage and a minimum water input 30 according to this process is bound to recirculating to the fermentor large amounts of stillage of the same concentration as the discharged stillage and the estab-



15

20

25

30

35

lishing of a high equilibrium concentration of nonfermentable substance in the fermentor, the success of the method can largely be derived to the surprising discovery that conventional yeast species were adaptable to give sufficient ethanol productivity despite the high concentration of salts and other dissolved non-fermentable material in the fermentor.

According to the Swedish patent application 7901738-0 the distillation step in the continuous ethanol process was modified so that the discharge of a further concentrated stillage was made possible. The distillation step for separating ethanol from the yeast-free liquor was divided into an evaporator section and a stripping section, the stillage stream recirculated in the process was discharged from the bottom of the evaporator section, that is from an intermediate distillation level in the distillation step, while the stillage to be discharged was withdrawn from the bottom of the stripping section. Among process improvements obtained through this modification, the most important advantage is that the concentrated stillage stream can be discharged from the distillation step simultaneously as there is obtained through recirculation of a stillage stream of lower dry substance content than the discharged stillage stream a lower equilibrium level as to the concentration of non-fermentable substance in the fermentor compared with the above said original process at the same process water input.

Ethanol can be produced from several different raw materials of vegetabilic origin, which either contains directly fermentable sugars or polysaccarides which through enzymatic degradation or other degradation procedures can be converted to fermentable sugar. In



10

addition to fermentable substance and soluble non-fermentable substance, these raw materials contain various amounts of non-fermentable solid constituents like fibres, shells, lignin etc. At hydrolysis and fermentation of starch raw material such as grain, these solid substances occur in such an amount that they would cause clogging problems in the continuous fermentation circuit, for example clogging of the nozzle openings of the nozzle centrifuges suitably employed for the yeast separation. Besides, the viscosity of the concentrated stillage streams is negatively effected and may limit the possibility to reach maximum dry substance content in the discharged stillage.

According to a previous Swedish patent application 7811826-2 this problem is solved by removing the fibres and other solid material directly from the raw material stream by straining and washing out remaining fermentable material from the solids by means of recirculated stillage. A disadvantage of this process, however, is that despite the use of a counter-current multi step washing requiring a relatively costly equipment, a complete recovery of the remaining fermentable substance from the solid material is not obtained.

25

30

According to the present invention there is suggested a process, which is characterized in that the solid material is fed to the fermentor together with a substrate and that the solid material is separated from the stream of fermentation liquor withdrawn from the fermentor before the fermentation liquor is sent to the yeast separation step and the successive ethanol separation step, the solid substance being discharged from the fermentation circuit.

35

By this process there is avoided that the solid mate-



10

15

20

25

30

35

rial reaches the yeast separator and the distillation plant, simultaneously as the content of fermentable material in the solids is very low since the concentration of fermentable material in the fermentor is maintained at a very low level, usually less than 0.5 % by weight. The removal of the solid material can be carried out by any straining device or similar equipment. Since the stream of fermentation liquor, which due to an ethanol content in the range of 3-7 % by weight necessarily must be large, a substantial part of the fermentation liquor will accompany the fibre phase if a rapid and efficient straining is to be carried out for example by means of a bow sieve. The liquor that accompanies the solid material will thus contain considerable amounts of yeast and ethanol.

According to a preferred embodiment of the invention, the rejected solids from said draining step are therefore washed with recirculated stillage in such an amount that a dilution of about 3 - 4 times is obtained. Then the wash liquor is separated off from the solid material in a suitable device, such as a centrifugal sieve, and is returned to the fermentor. Thereby the major parts of the remaining yeast and ethanol are washed out.

For the washing step also part of or all of the required process water input can be used. If for example the raw material is a grain product, the process water is usually added in the so called "mashing in step" and also in the form of direct steam in a successive hydrolysis step. Since this process water wholly or partly can be replaced by recirculated stillage, it is realized that the input of water to other parts of the process, in the present case to the washing step, will not effect on the total amount of water fed to the



process.

The solid substance stream strained off after the washing step and having a dry substance content in the range of 25-35 % by weight will still contain a slight amount of ethanol. Therefore, there is suggesed as a further preferred embodiment of the invention that the strained solid stream is subjected to further dewatering, for example by means of a press device, whereat something like half of the remaining ethanol can be pressed out with the press water and returned to the fermentor, while a press mass of a dry substance content of about 40 % can be discharged from the process.

15

20

25

30

35

10

5

By discharging the solid material from the process circuit before the yeast separation step and the distillation step, the possibilities to achieve a maximum concentration in the stillage discharged from the distillation step are improved. If the distillation step is designed according to the above said Swedish patent application 7901738-0 that is taking out a stillage stream at an intermediate level for recirculation and discharging stillage from the bottom of a stripping section, the present invention makes possible the production of a still more concentrated stillage stream from the stripper than before. If the solid material is passed through the yeast separating step or is drained off prior to the yeast separation step and rejoined with the feed to the stripper, the fluidity may be reduced in the bottom of the stripper so that the maximum upper dry maximum concentration of non-fermentable material in the fermentor cannot be fully utilized. Therefore, by separating off the solid material between the fermentor and the yeast separation step according to the invention, the limit for the dry substance con-



30

35

tent in the discharged stillage can be further raised to the range of 35-45 % by weight and the process water input can be further reduced.

5 For efficient recovery of all waste products at the process according to the invention, it is further suggested that the concentrated stillage from the distillation step is joined with the removed solid material, whereafter the mixture is dried in suitably an indi-10 rect heated drying plant. The steam dried off in the drying plant is reused in the process for heating purpose such as heating a stripping unit or a successive rectifying unit, whereat also thermocompression can be used to raise the energy level of the steam. Further 15 example of reusing the drying steam is as a heat source and process water in the substrate preparation step. As an alternative to thermo-compression, the drying can be carried out under overpressure so that drying steam of higher energy level is obtained di-20 rectly.

The invention will now be further described by means of a preferred embodiment of the same, whereat it is referred to the accompanied drawing showing a flow shart of a continuous ethanol fermentation process according to the invention.

A raw material stream 1 of suitable fine divided form, such as crushed corn, is fed to a substrate preparation step 2 comprising a liquification step and a saccharifying is fed to one part through a stream 3 of pure water and to another part through a stream 4 of recirculated stillage from the distillation step 5. From the substrate preparation step 2 the substrate stream 6 is fed into the fermentor 7, in which steady-state concentrations are maintained, the ethanol con-



10

15

20

25

30

35

centration being in the range of 3-7 % by weight and the concentration of fermentable substance being low, preferably less than 0.5 % by weight. From the fermentor a carbon dioxide stream 8 is continuously discharged.

From the fermentor a stream of fermentation liquor 9 is withdrawn. From the stream 9 fibres and other solid material are strained off in a strain device 10, from which a stream 11 of rejected material and a stream 12 impoverished of solid material are discharged. The stream 12 of fermentation liquor is conveyed to a centrifugal separator 13, from which there are discharged a heavier yeast suspension phase 14 which is recirculated to the fermentor 7 and a lighter essentially yeast-free phase 15 which is conveyed to the distillation unit 5. Before that the yeast-free stream 15 is heat exchanged in a heat exchanger 16 with a recirculated stillage stream 17 from the distillation unit 5.

The distillation unit 5 is divided into an evaporator unit 18 and a stripping unit 19. The yeast-free stream of fermentation liquor 15 is fed to the evaporator unit 18, discharging from the top of which ethanol enriched vapours which may be conveyed to an eventually successive rectifying step. From the bottom of the evaporator unit 18. representing an intermediate distillation level in the distillation unit 5, a stillage stream 20 is discharged, a larger part of which is recirculated in the form of said stillage stream 17 to the fermentation circuit and a smaller part 21 is fed to the stripping unit 19. From the stripping unit 19, which is indirect heated through 22, an overhead vapour stream 23 is discharged, which also contains the remaining ethanol of the stillage stream 21. The

vapour stream 23 is fed to the evaporator unit 18 as



25

direct heating medium. From the bottom of the stripping unit 19 a concentrated stillage stream 14 is discharged, the concentration of which, due to the elimination of solid material before the distillation step according 5 to the invention, may reach a dry substance content exceeding 30 % by weight, in some cases even exceeding 35 % by weight. The reject stream 11 from the straining device 10 is conveyed to a further straining step 25 being for example a centrifugal sieve device, in which the reject stream is simultaneously diluted and 10 washed with a stream 26, which is part of the recirculated stillage stream 17 after having been subjected to cooling in heat exchanger 16 and eventually further cooling in heat exchanger 27. In the straining device 15 25 a major part of the washing liquor is separated off and returned through 29 to the fermentor 7. The solid material strained off and having a dry substance content of for example the range 25-35 % by weight, is conveyed through 30 to a press device 31, in which a large part of the remaining liquor is pressed off and returned through 32 to the fermentor. The remaining press mass, now having a dry substance content of about 40 % by weight or higher, is discharged through 33, mixed with the stillage stream 24 and fed to a drying plant 34. From the drying plant 34 a dried product 35 is discharged, which can be utilized as animal feed stuff and having a protein content which may amount to about 30 % by weight.

30 As alternative washing liquor at the straining step 25 also pure water supplied to the process can be used, which in the figure has been shown by line 37. From the figure it can further be seen that the recirculated stillage stream 17 can be returned at different points and in various portions in the pro-35 cess circuit. As to stillage recirculation, in addi-



tion to said streams 26, i.e. washing liquor at the straining step 25, and the stream 4 for substrate preparation, stillage can of course be recirculated directly to the fermentor 7, which is shown by line 38.

5

10

15

20

25

30

In the specification and patent claims the term "solid substance" has been used for solid material of such a size that permits straining off by means of conventional straining devices. In this context it should be mentioned that distinct borders can hardly be drawn between particulate or fibrous substances, suspended finer material, colloidal material etc. The recirculated yeast fraction contains for example suspended yeast cells, and the other "yeast-free" and streams poor in "solid material" contain verious amounts of i.e. fine suspended and colloidal proteins. Besides these protein aggregates are subjected to a certain modification when passing through the distillation step. By being subjected to heat therein, coagulation occurs and larger aggregates are formed. When these larger aggregates through the recirculated stillage stream once again reach the straining step 10 and 25. their tendency to stay in the reject phase has increased, and consequently the process according to the invention also implies that the stripping unit is unloaded from part of these viscosity increasing aggregates, while still these for feeding purpose desirable proteins are recovered through remixing the sieve reject with stillage concentrate from the stripping unit.



Claims

- A process for the production of ethanol by continuous fermentation of a carbohydrate containing substrate (6) of a raw material which contains fermentable substance as well as non-fermentable soluble and solid substance, 5 wherein the substrate after eventually having been treated in a substrate preparation step (2) comprising for example hydrolysis of the raw material, is fermented in a fermentor (7), comprised in a continuous process circuit including the fermentor (7), a yeast separation 10 step (13) and a distillation step (5), a stream (9) of fermentation liquor is continuously withdrawn from the fermentor (7) and separated in said yeast separation step (13) into a yeast concentrate stream (14), which is continuously recirculated to the fermentor to maintain in the fermentor a concentration of active yeast cells corre-15 sponding to a certain ethanol productivity, and into an essentially yeast free stream (15), said yeast free stream (15) is continuously separated in said distillation step into an ethanol enriched stream (19a), which is discharged from the process circuit, and into at least one stillage stream (24, 17), part of said stillage stream (17) being continuously recirculated to the fermentor and/or to said substrate preparation step and the remaining part of said stillage'stream (24) being discharged from the process circuit, characterized in that the sub-25 strate fed to the fermentor contains said solid substance, and that said stream of fermentation liquor (9) is separated into a stream (11) enriched in solid substance, which is discharged from said process circuit, and into 30 a solid impoverished stream (12) which is fed to said yeast separation step (13).
 - 2. The process of claim 1, characterized in that said stream (11) enriched in solid substance is



10

15

20

25

washed by a stillage stream (26) which is recirculated in the process circuit and having been cooled, e.g. through heat exchange with said yeast-free flow (15), to a temperature not harmful to the yeast cells which remain in said stream enriched in solid substance, and/or with part of the required water input (37) to the process circuit, whereafter the washed solid substance is separated e.g. in a straining step (25) into an essentially yeast-free solid substance stream (30), which is discharged, and a wash liquor stream (29), which is returned to the fermentor.

- 3. A process according to claim 2, c h a r a c t e r i z e d i n that said stream enriched in solid substance (ll) is washed with at least part of a first stillage stream (17), which is withdrawn from an intermediate distillation level in the distillation step (5) and recirculated into said process circuit, and that a second stillage stream (24) of higher dry substance than said first stillage stream (17) is discharged as a bottom product from said distillation step (5) and removed from said process circuit.
- 4. A process according to any of the preceding claims, c h a r a c t e r i z e d i n that said yeast-free solid substance stream (30) is subjected to further concentrating, e.g. in a pressing step (31), thereat being separated into a concentrate stream (33) and a liquor stream (32) which is returned to said process circuit, suitably to the fermentor (7).
- 5. A process according to any of the preceding claims,
 30 c h a r a c t e r i z e d i n that a first stillage
 stream (17) is discharged at an intermediate distillation level in the distillation step (5) and recirculated
 in said process, and that a second stillage stream (24)



with a dry substance content of more than 35 % by weight is discharged as bottom product from said distillation step (5) and is removed from said process circuit.

- 6. A process according to any of the preceding claims,
 5 c h a r a c t e r i z e d i n that said yeast-free solid substance stream (30) or said concentrate stream (33) is dried in a drying step (34) together with a stillage stream (24) discharged as a bottom product in the distillation step (5) to obtain a dried product
 10 (35) usable as feed stuff.
- 7. A process according to claim 6, c h a r a c t e r i z e d i n that steam produced in the drying step (34) is used after eventual thermo-compression as a heating medium in said process circuit, for example as heating medium in the distillation step (5) and/or in the substrate preparation step (2).



AMENDED CLAIMS

[received by the International Bureau on 08 April 1983 (08.04.83); original claims 1 to 7 cancelled; new claims 1 to 6 follow]

A process for the production of ethanol by continuous fermentation of a carbohydrate containing substrate (6) of a raw material which contains fermentable substance as well as non-fermentable soluble and solid substance, wherein the substrate after eventually having been treated in a substrate preparation step (2) comprising for example hydrolysis of the raw material, 10 is fermented in a fermentor (7), comprised in a continuous process circuit including the fermentor (7), a yeast separation step (13) and a distillation step (5), a stream (9) of fermentation liquor is continuously withdrawn from the fermentor (7) and separated in said 15 yeast separation step (13) into a yeast concentrate stream (14), which is continuously recirculated to the fermentor to maintain in the fermentor a concentration of active yeast cells corresponding to a certain ethanol productivity, and into an essentially yeast free 20 stream (15), said yeast free stream (15) is continuously separated in said distillation step into an ethanol enriched stream (19a), which is discharged from the process circuit, and into at least one stillage stream (24, 17), part of said stillage stream (17) being con-25 tinuously recirculated to the fermentor and/or to said substrate preparation step and the remaining part of said stillage stream (24) being discharged from the process circuit, characterized the substrate fed to the fermentor contains said solid 30 substance, and that said stream of fermentation liquor (9) is separated into a stream (11) enriched in solid substance and into a solid impoverished stream (12) which is fed to said yeast separation step (13), that said stream (11) enriched in solid substance is washed 35 by a stillage stream (26) which is recirculated in the



30

35

process circuit and having been cooled, e.g. through heat exchange with said yeast-free flow (15), to a temperature not harmful to the yeast cells which remain in said stream enriched in solid substance, and/or with part of the required water input (37) to the process circuit, whereafter the washed solid substance is separated e.g. in a straining step (25) into an essentially yeast-free solid substance stream (30), which is discharged, and a wash liquor stream (29), which is returned to the fermentor.

- A process according to claim 1, character terized in that said stream enriched in solid substance (11) is washed with at least part of a first stillage stream (17), which is withdrawn from an intermediate distillation level in the distillation step (5) and recirculated into said process circuit, and that a second stillage stream (24) of higher dry substance than said first stillage stream (17) is discharged as a bottom product from said distillation step (5) and removed from said process circuit.
- 3. A process according to any of the preceding claims, c h a r a c t e r i z e d i n that said yeast-free solid substance stream (30) is subjected to further concentrating, e.g. in a pressing step (31), thereat being separated into a concentrate stream (33) and a liquor stream (32) which is returned to said process circuit, suitably to the fermentor (7).
 - 4. A process according to any of the preceding claims, c h a r a c t e r i z e d i n that a first stillage stream (17) is discharged at an intermediate distillation level in the distillation step (5) and recirculated in said process, and that a second stillage stream



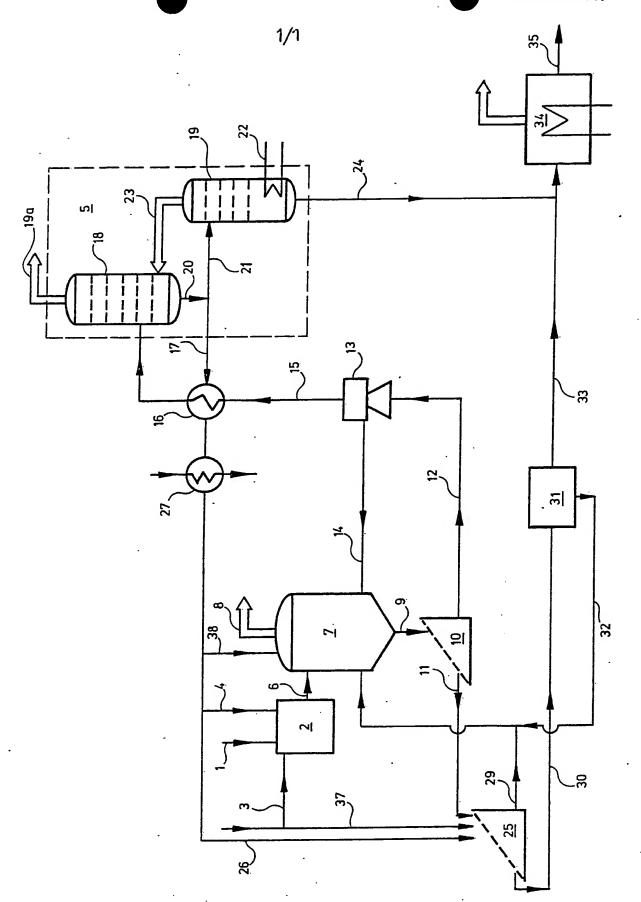
(24) with a dry substance content of more than 35 % by weight is discharged as bottom product from said distillation step (5) and is removed from said process circuit.

5

10

- 5. A process according to any of the preceding claims, c h a r a c t e r i z e d i n that said yeast-free solid substance stream (30) or said concentrate stream (33) is dried in a drying step (34) together with a stillage stream (24) discharged as a bottom product in the distillation step (5) to obtain a dried product (35) usable as feed stuff.
- 6. A process according to claim 5, characte15 rized in that steam produced in the drying step
 (34) is used after eventual thermo-compression as a
 heating medium in said process circuit, for example as
 heating medium in the distillation step (5) and/or in
 the substrate preparation step (2).







INTERNATIONAL SEARCH REPORT

International Application No PCT/SE82/00369

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) ! According to International Patent Classification (IPC) or to both National Classification and IPC 3

C 12 P 7/06

II. FIELDS SEARCHED

Minimum Documentation Searched 4

Classification Symbols Classification System C 12 B 3/04-10, 1/00, C 12 C 11/14 IPC 1,2 IPC:3 C 12 P 7/06-12

> Documentation Searched other than Minimum Documentation to the Extent that such Documents are included in the Fields Searched 6

SE, DK, NO, FI classes as above

Category *	Citation of Document, 18 with I	ndication, where appropriate, of the relevant passages 17	Relevant to Claim No. 15
x	31 Ju	133-5 (ALFA-LAVAL AB) uly 1979 A1, 2 903 273	
X	1,7 Ma	315 (ALFA-LAVAL AB) ay 1980, especially claim 2 Al, 2 946 161	
х	27 Au	738-0 (ALFA-LAVAL AB) ugust 1980 Al, 3 007 138	
A		9 518 (UTIKAL HANS) ovember 1980	
	•		
			. •

- Special categories of cited documents: 18
- document defining the general state of the art which is not considered to be of particular relevance
- earlier document but published on or after the international filing date
- document which may throw doubts on priority claim(s) or which is cited to establish the publication date of enother citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or
- document published prior to the international filing date but later than the priority date claimed
- later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step
- document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- "&" document member of the same patent family

Date of Mailing of this International Search Report 5

IV. CERTIFICATION

Date of the Actual Completion of the International Search

. 1983 **-**02- 08

International Searching Authority 1

Swedish Patent Office

Signature of Authorized Officer 20

Britta Samuelsson

1983-02-02

This Page is Inserted by IFW Indexing and Scanning Operations and is not part of the Official Record

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

BLACK BORDERS

IMAGE CUT OFF AT TOP, BOTTOM OR SIDES

FADED TEXT OR DRAWING

BLURRED OR ILLEGIBLE TEXT OR DRAWING

SKEWED/SLANTED IMAGES

COLOR OR BLACK AND WHITE PHOTOGRAPHS

GRAY SCALE DOCUMENTS

LINES OR MARKS ON ORIGINAL DOCUMENT

REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY

IMAGES ARE BEST AVAILABLE COPY.

□ OTHER: _____

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.